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Indian Standard

METHODS FOR COUNTING AND SIZING PARTICLES IN INSULATING LIQUIDS

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

METHODS FOR COUNTING AND SIZING PARTICLES IN INSULATING LIQUIDS

NATIONAL FOREWORD

This Indian Standard, which is identical with IEC Pub 970 (1989) 'Methods for counting and sizing particles in insulating liquids', issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of Fluids for Electrotechnical Applications Sectional Committee (ETD 03), and approval of the Electrotechnical Division Council.

In the adopted standard certain terminology and conventions are not identical with those used in Indian Standards, attention is specially drawn to the following:

a) Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'Indian Standard'.
b) Comma (,) has been used as a decimal marker while in Indian Standard, the current practice is to use a point (.) as the decimal marker.

In the adopted standard, reference appears to an International Standard for which Indian Standard also exists. The corresponding Indian Standard which is to be substituted in its place is listed below along with its degree of equivalence for the edition indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
IEC Pub 475 (1974) Method of sampling liquid dielectrics	IS 6855 : 1973 Method of sampling for liquid dielectrics	Technically equivalent

The concerned technical Committee has reviewed the provisions of ISO 4402, ISO/DIS 4407 and ISO/DIS 4408 referred in the adopted standard and has decided that they are acceptable for use in conjunction with this standard.

Only the English language text in the International Standard has been retained while adopting it in this Indian Standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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SECTION ONE - GENERAL

1. Scope

This standard describes sampling procedures and methods for determinating particle concentration and particle size distribution. Three methods are recommended, one using an automatic particle size analyzer working on the light interruption principle and two based on the counting, with an optical microscope, of particles on the surface of a filtering membrane with transmitted or incident light. The latter two methods are described in ISO/DIS 4407 and ISO/DIS 4408. All methods are applicable both to unused liquids and to liquids in service.

Appendix A contains an alternative sampling procedure using a syringe and Appendix B describes three methods for the calibration of automatic counters.

Notes *1.-* The methods are not intended to measure particulate matter in liquids containing sludge.

2.- The methods specified are only applicable to measurements related to a limited range of size and number.

2. Reference documents

IEC publication:

475 (1974):	Method of sampling liquid dielectrics

ISO standard:

4402 (1977):	Hydraulic fluid power - Calibration of liquid automatic particle-count instruments - Method using Air Cleaner Fine Test Dust contaminant.

Draft International standards:

ISO/DIS 4407:	Hydraulic fluid power - Fluids - Determination of solid particle contamination - Counting method using a microscope under transmitted light.
ISO/DIS 4408:	Hydraulic fluid power - Fluids - Determination of solid particle contamination - Counting method using a microscope under incident light.

3. Significance

It is well known that particles have a detrimental effect on the dielectric strength of insulating liquids. It has long been the practice to include in specifications of insulating liquids the requirement that the fluid be clear and free of visible particulate matter. However, there has been no standard method for quantitative estimates, so that practices have differed. This standard provides standard procedures for the test.

Filtration of insulating liquids is an established practice in the electrical industry. The procedure described may serve to assess the performance of the filter system. The results of the measurements depend on the method used. With the automatic counter the measured values also depend on the calibration procedure and in particular on the calibration substance (fine test dust or spherical standards).It is therefore essential that the methods of analysis and calibration are specified when quoting results.

The particle content of a sample may depend on the sampling point, time elapsed since filling, oil circulation flow rate, and/or the time an oil is left to stand. Circulation of an oil filling will lead to more consistent particle count results.

With aged liquids, oil oxidation products which are dissolved at operating temperatures may precipitate under conditions of prolonged stand time at room temperature. This process, which depends on the length of stand time, the storage temperature and the ageing condition of the liquid, may have an effect on the particle count determination.

Storage of the samples will generally lead to a sedimentation of particles. Fine particles may be also coalesce to form larger particles. Unless all the contents of the sampling vessel are analyzed in one single test, shaking will be necessary.

This can be done manually, in a shaking machine, or by ultrasonic treatment. If high-energy ultrasonic treatment or higher shear mixing is used, there is a risk that the particle count determined will be enhanced owing to breakdown of sludge and other large particles.

4. Comparison and limitations of the methods

Automatic particle counters using the light interruption principle are quick and easy to use, but the following points should be borne in mind:

1) With some liquids it may be necessary to modify their viscosity to comply with the instrument's running parameters.

2) It is necessary to choose a sensor head suitable for counting in the size range required. No single head can count both very small particles (<2 µm) and very large particles (>200 µm).

3) The instrument records the light interruption area of the particle and from this calculates the diameter of a sphere having the same area (or the longer axis of a specified ellipsoid with the same area).

   This means that no information on the shape of the particles is obtained, which especially constitutes a limitation where the recognition of fibres is concerned. Their narrow and elongated shape results in a slight light obturation and consequently in a very small equivalent sphere diameter. When it is important to evaluate the concentration of fibres, automatic counters cannot perform this task adequately.

4) When air-saturated or over-saturated liquids are shaken manually or in a shaking machine, or given high-energy ultrasonic treatment, or treated at full vacuum, finely dispersed microbubbles may be formed in the liquid. In the optical system of the automatic counter, these microbubbles will be counted as solid particles.

   These difficulties are avoided when using optical microscopic methods. In addition, optical microscopy may give some information about which types of particles are present. These methods are, however, much more time-consuming and operator-dependent and it may often be very difficult to count particles of less than 5 µm.

5. Types and identification of particles

Particles found in an insulating liquid may be metal-working residues in the form of iron, copper, brass and aluminium particles, welding cinder and sandblasting material. Metal particles may also originate from pump wear. Other types of particles include soot and sludge from arcing in or ageing of the liquid, cellulose lint, sand, dust, and particles of varnish, plastic or rubber.

A knowledge of which particle types are present in the insulating liquid can, in certain cases, help in assessing the condition of the equipment or in diagnosing a fault. Certain particles may be identified by filtering a liquid sample through a membrane filter and examining the residue under a microscope.

Some fibres can be identified with specific identification stains, and a number of metals by means of spot tests or microscopic chemical methods. Metallic materials may also be identified and quantified by instrumental methods and wet chemical analysis. A detailed description of methods for the identification of particles is, however, outside the scope of this standard.

6. Sampling

6.1 *General remark*

The sample taken should only be used for the particle count determination

6.2 *Sampling vessels*

The sampling vessels recommended in this standard are cylindrical, flatbottomed, wide-necked, clear glass bottles fitted with a polypropylene threaded cap forming a seal with the bottle without the use of any inserts.

When automatic particle size analyzer is used, the volume of the bottle shall be between 150 cm³ and 500 cm³.

For microscopy, the bottles shall have at least 100 cm³ capacity and be permanently marked to indicate 100 cm³ sample size.

An alternative method for oils in service using syringes as sampling vessels is given in Appendix A.

6.3 *Cleaning of sampling bottles*

It is recommended that the bottles be cleaned to achieve a blank count of less than 200 particles above 5 µm per 100 cm³. The test should be performed on the filtered solvent used in the last stage of the cleaning process.

A cleaning method is indicated for guidance, but other methods can be used provided they have at least the same effectiveness.

a) Wash with warm water containing detergent

b) Rinse with warm water and drain

c) Rinse thoroughly with 0.45 µm membrane filtered acetone to remove water

d) Rinse with 0.45 µm membrane filtered petroleum ether or with another suitable solvent. Leave 1 cm³ or 2 cm³ of solvent in the bottle and close the bottle.

If ultrasonic agitation is used before counting particles, the cleaning procedure shall include ultrasonic treatment. Plunge the sampling bottle filled with 0.45 µm membrane filtered acetone in an ultrasonic bath for 15 min in place of step *c)* above

A residue of solvent in the bottle creates a positive pressure in the bottle helping to prevent contamination from the atmosphere when opening the bottle.

Warning.- Attention is called to national regulations associated with the use of solvents.

6.4 *General directions for sampling*

It is difficult to obtain representative samples from a drum. If sampling is found to be necessary, the procedure given in Clause A5 of Appendix A may be used or, alternatively, the procedures given in IEC 475. In the case of sealed power transformers and instrument transformers or similar equipment with small liquid volume, the manufacturer's instructions on sampling procedure and quantity shall be followed. A sample from a transformer should preferably be taken during oil circulation or immediately afterwards. The analysis obtained may depend on the sampling point selected. Confirmatory or follow-up samples should therefore always be taken from the same point.

Every precaution shall be taken when sampling not to contaminate the sample.

Outdoor sampling of insulating liquids in rain, fog, snowfall or high wind is only permitted if all precautions have been taken to avoid contamination of the samples. In this special case the use of a cover is necessary.

Ensure that sampling is done by an experienced person.

6.5 *Sampling procedure*

- Prior to taking the sample, the exterior of the sampling valve and the adjacent parts shall be thoroughly cleaned using lint-free wiping materials.
- Connect a length of a suitable plastic tube (resistant to the liquid sampled) to the sampling valve.
- If the sampling bottle contains filtered solvent, it may be necessary at low ambient temperature to warm the bottle with the hands, in order to create a positive pressure and thereby prevent the ingress of atmospheric particulate matter.

  Warning.- Petroleum ether, as well as many of its alternatives, is a highly flammable solvent, and appropriate safety measures must be observed when the bottle is opened.

  Thoroughly flush the valve and line by draining a sufficient quantity of liquid (e.g. 5 l) into a waste container.

Remove the cap from the bottle; do not empty out the solvent. Without interrupting the flow from the valve, substitute the bottle for the waste container and collect the required volume of sample as quickly as possible. Then remove the sample bottle and replace the waste container.

- Replace the bottle cap without overtightening.
- Close the sampling valve and replace any protection. Label the sample.
- The sample shall be protected against light during transportation and storage.

6.6 *Labelling of samples*

6.6.1 *Samples from tanks*

Labels shall carry the following markings:

- tank identification;
- sampling point;
- type of insulating liquid;
- date of sampling.

6.6.2 *Samples from electrical equipment*

Labels shall carry the following markings:

- equipment identification;
- sampling point;
- equipment in factory/in service;
- liquid identification;
- temperature of liquid;
- date of sampling.

7. Preparation of the samples for analysis

Samples should be processed as soon as possible.

The effects of storage of samples are described in Clause 3. The samples can be mixed by shaking (manually or in a shaking machine) or by ultrasonic treatment. If high-energy ultrasonic treatment is used, however, there is a risk that the particle number is enhanced owing to breakdown of large particles, and also that finely dispersed microbubbles are formed in the liquid. As in the optical system of the automatic counter such microbubbles will be counted as particles a vacuum treatment is necessary (see Sub-clause 8.6.2). When too high a vacuum is applied to air-saturated or over-saturated liquids, however, microbubbles may be released from the liquid.

Note.- It is advisable that the agitation and vacuum procedure developed be tested for effectiveness. This can be done by taking several samples simultaneously. One sample is tested immediately after sampling, with no agitation or vacuum treatment at all, another after treatment and a third after some storage time. The test should be applied to liquids with a varying degree of particle content, state of ageing, and air content.

SECTION TWO - METHOD A

8. Counting and sizing of particulate matter in insulating liquids using an automatic particle size analyser

8.1 *Summary of method*

The sample is agitated to suspend the particles, then passed at an optimum flow rate through the sensor unit of the particle counter. After the required fluid volume has been passed through the sensor the count is terminated and the results recorded.

8.2 *Apparatus and auxiliary materials*

8.2.1 An automatic particle counter fitted with a sensor operating on the light interruption principle and suitable for counting within the range of 2.5 µm to greater than 100 µm.

The automatic particle counter shall be capable of:

a) Sorting particles in at least five size ranges.

b) Providing a specific particle count and distribution for a measured quantity of reference material suspended in 100 cm^3 of a suitable liquid to a precision of ±10% of the total count. To obtain sufficient accuracy, the number of particles counted shall be greater than or equal to 200 in the smallest size ranges.

c) Not saturating (i.e. provide a lower than expected count as a result of particle coincidence within the sensing zone) when analyzing a suspension containing less than 2 000 particles per cubic centimeter of fluid.

Note.- Alternative detectors may be used to extend the size range of the particles measured.

8.2.2 Compressed gas - a pressurized source of air or nitrogen free from oil or water contamination filtered through a 0.45 µm membrane filter. The capacity, pressure range and constant pressure controls should meet the requirements of the particular equipment in use.

8.2.3 Vacuum pump capable of supplying a steady vacuum.

8.2.4 Solvent dispenser fitted with a 0.45 µm membrane filter at the outlet.

8.2.5 Solvent - petroleum ether with a boiling-range of 40 °C to 60 °C, or suitable alternative filtered through a 0.45 µm membrane filter.

8.2.6 Detergent - liquid, water soluble, commercial grade.

8.2.7 Calibration standard - one of the following:

- Latex spheres of known numbers and size ranges dispersed in a suitable liquid.
- A set of mono-sized latex spheres dispersed in a suitable liquid.
- Air Cleaner Fine Test Dust (A.C.F.T.D.).

8.2.8 Optional: ultrasonic cleaning bath (see Clause 7).

8.3 *Calibration procedures*

8.3.1 The calibration of the instrument shall be carried out using one of the procedures given in Appendix B.

8.3.2 Calibration shall be carried out annually or when any repair/change is made to the sensor or whenever results are suspect.

8.4 *Preparation of the apparatus for counting*

Check that the instrument is set to the calibration numbers for the required size ranges in accordance with the procedure given in the instrument manual.

When the instrument has been switched off, the check procedure shall be carried out prior to use or daily, whichever is less frequent

If the instrument is permanently switched on, the check procedure shall be carried out at least monthly.

8.5 *Preparation of sample before counting*

8.5.1 Remove any visible contamination from the exterior of the bottle.

8.5.2 Samples which are found on visual examination to contain water or suspended solids (e.g. sludge) likely to affect the performance of the sensor shall be rejected.

8.5.3 Sample dilution should be avoided as far as possible. However, if dilution is necessary in order to comply with the instrument run parameters, a dilution with a liquid of the same type as the sample (or with a compatible solvent), filtered through a 0.45 µm membrane filter, may be used. Then record the dilution ratio.

Note.- In order to meet the instrument run parameters, viscosity modification of the liquid may be achieved by heating. If recourse to this is necessary, due regard to the instrument manufacturer's recommendations regarding sensor life should be noted. It should also be established that the precision obtained is acceptable.

8.6 *Preparation of sample for counting*

8.6.1 Mix the prepared sample either by shaking gently by hand for 1 min and/or by immersing the bottle in an ultrasonic cleaning bath.

8.6.2 If necessary, apply a moderate vacuum for at least 1 min to remove air bubbles.

8.6.3 Flush the sensor with a small amount of the test sample.

8.6.4 Count the test sample wherever possible from the sample bottle as received. If the counting is not commenced within 2 min repeat procedures 8.6.1 and 8.6.2.

Note.- It is advisable, when using automatic bottle sampling apparatus, that the sensor uptake tube be filled with the test fluid before applying the vacuum.

8.7 *Counting procedure*

8.7.1 Ensure that the instrument has been switched on long enough to become stabilized before counting is initiated.

8.7.2 The counting procedure shall be carried out in accordance with the instrument manufacturer's operating instructions, paying particular attention to the required flow rate and selecting the differential (delta) mode of counting. The cumulative mode may be used if a delta mode is not available on the instrument.

8.7.3 The sample shall be counted in at least two equal volumes, greater than or equal to 20 cm^3. These two counts should agree within 10% in the smallest size range. If this requirement is not attained the sample shall be discarded or re-agitated and re-counted. It is desirable that in each size range the sum of the two counts should be not less than 200. In the event of there not being sufficient particles to reach a total count of 200 in any size range, report the actual number counted as well as the concentration.

Note.- The above minimum count of 200 ensures that the counting accuracy is within ±10% with a 95% confidence.

8.7.4 After each sample has been counted remove the sample bottle and flush the system with solvent, filtered through a 0.45 µm membrane filter. It is not recommended that sensor be dried out after flushing.

8.7.5 Calculate the particle concentration in each size range from the mean of the counts obtained for each sample aliquot, taking into account the volume of the aliquot and the dilution ratio.

8.8 *Report*

Report the number of particles per 100 cm^3 of the original sample in at least the following size ranges (see note):

Particle size range (p)

Micrometres (µm)

$2.5 < p \leq 5$
$5 < p \leq 15$
$15 < p \leq 25$
$25 < p \leq 50$
$50 < p \leq 100$
$p > 100$

Total number of particles > 2.5
Total number of particles > 5
Total number of particles > 15

The report shall also include the following:

- method of calibration;
- method of agitation;
- date of sampling;
- date of analysis.

Note.- For liquids in service, it may be sufficient to report just the number of particles greater than 5 µm and greater than 15 µm.

8.9 *Precision*

The repeatability and reproducibility of this method have not yet been established.

SECTION THREE - METHOD B

9. Counting and sizing of particulate matter in insulating liquid using optical microscopy

9.1 *Principle*

A known volume of insulating liquid is filtered under vacuum conditions through a membrane filter to collect contaminants on the filter surface. The membrane is then mounted between glass slides and examined microscopically by transmitted or incident light to measure, count and size particles according to their largest dimension.

9.2 *Procedure by transmitted light*

Counting and sizing of particulate matter microscopically by transmitted light shall be made according to the procedure described in ISO/DIS 4407.

9.3 *Procedure by incident light*

Counting and sizing of particulate matter by incident light shall be made according to the procedure described in ISO/DIS 4408.

APPENDIX A

USE OF SYRINGES AS SAMPLING VESSELS

A1. *Type of syringe*

Syringes shall be made of polypropylene or other suitable polymeric materials and have a volume of at least 150 cm^3 when using automatic counters. For microscopy, the syringe capacity shall be at least 100 cm^3.

Note.- For automatic counters an alternative is to use three syringes of 50 cm^3 capacity per sample. This will, however, reduce the accuracy of the result. For liquids with a high particle content one syringe with 50 cm^3 capacity may suffice.

A2. *Cleaning of syringes*

The same procedure as for bottles (Sub-clause 6.3) can be used, except that no solvent shall be left in the syringe.

A3. *General directions for sampling*

The same considerations given in Sub-clause 6.4 apply here.

A4. *Sampling from apparatus or tank with sampling valve*

- Prior to taking the sample, the exterior of the sampling valve and the adjacent parts shall be thoroughly cleaned with lint-free wiping materials.
- Connect the sampling valve directly to a two-way cock with a plastic tube resistant to the liquid.
- Thoroughly flush the sampling valve, the tube and the two-way cock by draining a sufficient quantity of liquid into a waste container.
- Connect the syringe directly to the two-way cock and take the sample.
- Disconnect the syringe and plug its bottom.
- Close the sampling valve and replace any protection.
- The sample shall be protected against light during transportation and storage.

A5. *Sampling from tank or drum without bottom valve*

- Connect a two-way cock to a plastic tube resistant to the liquid.

- Plunge the tube into the container.

- Connect a syringe to the two-way cock.

- Suck a sufficient quantity of liquid to rinse the system, by repeatedly filling and emptying the syringe.

- Replace the syringe with a new one and take the sample.

- Disconnect the syringe and plug its bottom.

- The sample shall be protected against light during transportation and storage.

A6. *Labelling of samples*

See Sub-clause 6.6.

A7. *Preparation of the samples for analysis*

See Clause 7.

APPENDIX B

CALIBRATION OF LIQUID AUTOMATIC PARTICLE COUNTERS

Three methods for calibration of the instrument are described.

Note.- An approximate conversion can be made between spherical and A.C.F.T.D. calibration by multiplying the figure obtained using spherical calibration by 1.54. To convert from A.C.F.T.D. to spherical, multiply by 0.65.

B1. *Calibration using A.C.F.T.D. (Air Cleaner Fine Test Dust)*

Calibration with A.C.F.T.D. shall be accomplished according to ISO 4402. The procedure given in ISO 4402 may be used to determine saturation levels for the counter.

B2. *Calibration using mono-sized calibrated latex spheres*

B2.1 A suspension of known mono-sized latex spheres shall be prepared in a compatible 0.45 µm filtered liquid. The size of the latex spheres shall correspond to the lower end of the sensor range.

B2.2 Prepare the suspension in the same way as described in Sub-clause 8.6.1.

B2.3 Count the calibrating suspension at the manufacturer's recommended flow rate with the threshold level of the first channel set so that all the spheres are counted in a narrow Gaussian distribution. The flow rate shall be maintained for all counting unless the instrument is recalibrated under another flow rate condition.

B2.4 Adjust the second channel's threshold setting until a half-count of the first channel is obtained.

B2.5 Determine the calibration (voltage) number of the second channel's threshold setting. This number represents the mean size of the mono-spheres.

B2.6 Repeat points B2.1 to B2.5 for at least four larger calibrated mono-sized latex spheres within the range of the sensor head.

B2.7 Plot a graph of mean particle size against voltage setting using log - log paper as follows:

a) Use the horizontal axis for calibration setting and the vertical axis for particle size for each channel threshold.

b) Plot calibration setting against particle size for each channel threshold.

c) Draw a smooth curve through the points and read off the calibration setting corresponding to the required size ranges on the graph by interpolation.

Note.- Linear regression may be used to obtain the best linear calibration graph when this technique is applied to automatic particle counters which incorporate a.c. amplifiers.

B3. *Calibration using a known distribution of latex spheres*

B3.1 Set the first channel of the automatic particle counter at its smallest particle size setting and fix the other channels at progressively higher calibration numbers as recommended by the manufacturer's instructions.

B3.2 A suspension of a known distribution of latex spheres shall be prepared in a compatible 0.45 µm filtered liquid.

B3.3 Prepare the suspension in the same way as described in Sub-clause 8.6.1.

B3.4 Introduce the calibration solution into the apparatus.

B3.5 Adjust the flow through the sensor to the manufacturer's recommended flow rate. This flow rate shall be maintained for all counting unless the instrument is recalibrated under another flow rate condition.

B3.6 Count an accurately measured volume of suspension, using the manufacturer's instructions. Record the count.

B3.7 Adjust each channel threshold setting by altering the potentiometer controls in accordance with the instrument manual.

B3.8 Repeat until the counts for the specified channel are within the following percentage of the count for the calibration standard.

Size range:	up to and including 15 µm:	±5%
	above 15 µm up to 50 µm:	±10%
	above 50 µm:	±25%

B3.9 Once the channels thresholds are set within acceptable limits do not change them. Determine the calibration numbers for each channel threshold setting as recommended by the manufacturer.

B3.10 Prepare a graph in the same way as described in Sub-clause B2.7.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :		Telephone
Central :	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	311 01 31 331 13 75
Eastern :	1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern :	SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
Southern :	C. I. T. Campus, IV Cross Road, MADRAS 600113	235 02 16
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